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STUDY OF INFRARED ABSORPTION SPECTRA OF SOLIDS BY
THE POWDER METHOD. (ACETATES, FORMATES,
OXALATES, ACETYLACETONATES, SULPHATES).Messrs. Jean Lecomte and René Freymann,
Presented by Mr. Aimé Cotton.

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(Acétates, formiates, oxalates,
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STUDY OF INFRARED ABSORPTION SPECTRA OF SOLIDS BY
THE POWDER METHOD. (ACETATES, FORMATES,
OXALATES, ACETYLACETONATES, SULPHATES).

Messrs. Jean Lecomte and René Freymann,
Presented by Mr. Aimé Cotton.

ABSTRACT. The study of the spectra of powdered solids is shown to be as easy as the study of liquid spectra. The powder is spread on sheets of rock salt or potassium bromide and studied between 6 and 20 μ . This method avoids the danger of melting and facilitates the examination of insoluble and opaquely soluble compounds. The powder method with infrared absorption techniques presents a very extensive research area in all types of chemistry.

Whereas the study, between 6 and 20 μ , of infrared absorption spectra of liquids and gases has been especially developed, there has been up until the present time very little research in this spectrum field on solid substances in the powdered state. We shall endeavor to show here that this study can be carried out, between 6 and 20 μ , with the same ease as the examination of liquids. /1401¹

Following a method similar to the one used with ultraviolet [1], the substance studied is finely ground to mortar; then, after passage through a fine screen, it is spread out on a thin sheet of rock salt or potassium bromide. In the case of hygroscopic substances, a thin layer is deposited on the sheet of rock salt by the rapid evaporation of a concentrated alcohol solution of the substance under study. The absorption spectra were recorded using the equipment previously described by one of us [2]. /1402

This method for studying powders does not therefore offer any special difficulty. It has, moreover, no new features and several authors, including one of us, have used it [3]. It is remarkable that, in spite of its simplicity, this method has not been used more between 6 and 20 μ .

¹Numbers in the margin indicate pagination in the foreign text.

It allows examination of substances which would be decomposed by melting. It may likewise be applied to insoluble substances or those substances soluble only in water which is opaque in the middle infrared. The following table shows that some compounds, containing water of crystallization, can even be studied in this way. The application of the powder method using infrared absorption techniques certainly offers a very extensive field of research, as much in inorganic chemistry as in organic or biological chemistry.

Among the various compounds which we have examined by this method, we shall point out here various metallic salts, acetates, formates, oxalates, and acetylacetonates. Although we featured the examination of the acetates, we intend to also examine other substances. Therefore, we provide the following table to show the vast capabilities of application of the powder method. We refer, for a more detailed study, as well as for the origin of the bands observed, to a previous report.

In addition, we draw special attention to the considerably differing findings produced for the sulphates of copper when anhydrous and with 5 mol of water. We likewise point out that the small lines $705\text{-}806\text{-}861\text{ cm}^{-1}$ are undoubtedly owing to the combination of the frequencies of the crystalline lattice with the frequency 620 cm^{-1} of SO_4 .

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TABLE I

Acetates

Crystallized; usually containing one or several water molecules

CH ₃ COOH					888	935		1012	1045		1256	1395	1497		1668
Li				720	804	851	933		1025	1045	1127		1427	1555	
Gl	660		714	784				979	1030	1055	1114		1466	1575	
Na	923		1018	1047	1133		1408	1527	1613
Mg	624	664		720	804	847	942		1020	1053	1124		1422	1515	1658
K	922	968	1013	1046	1120		1414	1543	1618
Ca	614	668		711			930		1012	1050	1117		1408	1508	
Cu	631		692	719					1031	1120			1420	1552	
Zn	620		689	719			949		1013	1050	1124		1427	1520	
Ba	617	650		716			927		1012	1050	1107		1408	1522	1645
Pb	616	662		721			936		1017	1047	1124		1416	1522	1645
CO ₂		664	689	717	796		936		1013	1050	1117		1393	1490	1645

Formates

Na	684		772			1055	1121		1335		1550	
Cu	684	765	785				1121		1316		1543	
Pb	685	758	783						1321		1513	

Oxalates

(COOH) ₂ ·2H ₂ O		681	717					1016		1117	1218		1412	1515	1631
Ca		658	686		780	833	875	942	1021	1105		1307		1592	
Fe II	711		811				1117	1272		1439	1515	1592
Co		662	682	717	742	818		1031		1112		1323		1538	1613
					765							1370			
Pb		664		720	767	831		1016		1117	1238		1406	1520	

Acetylacetonates

Sc	770	794	821	922	1010	1101	1238	1361		1515	1645
										1153					
Fe III	766	797		920	1005	1111	1247	1353		1513	1689
Ni	766			930	1016	1082	1247		1412	1475	1610
										1174					

Copper Sulphate

Anhydrous.....		705	806	861	956		1076		1174						
5H ₂ O		990	1063	1101	1170						

Heavy type, intense or medium heat

Ordinary type, very weak or

doubtful band

Italic type, weak band

.....area not studied